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July 23, 2016

Mark Loeb
Quality Assurance Manager
TestAmerica, Inc. –Canton
4101 Shuffel Drive NW
North Canton, OH 44720

Dear Mark,

Please find enclosed the final findings report for the technical project audit performed at the TestAmerica-Canton facility on June 1 and 2, 2016. This report summarizes the findings of the audit and lists the specific corrective actions suggested to resolve the deficiencies identified. These deficiencies were identified and discussed with laboratory personnel during the exit briefing on June 2.

The laboratory has been given an opportunity to respond, and a number of issues have been addressed through email and phone conversations; however a formal audit response has not yet been received. Suggested corrective actions that remain outstanding are listed within this report. Completion of corrective actions which remain outstanding will signal closure of this project audit.

The laboratory has requested a "kick-off" meeting to discuss details not captured by the QAPP, particularly those pertaining to communications between the field and the laboratory. The auditor concurs that this meeting should be held, and that the agreements should be documented both in laboratory internal records (Project Notes) and in a shared Field-Laboratory Coordination Memorandum, which should be added to the QAPP as an attachment immediately following the meeting.

We are grateful for the cooperation and assistance provided by all laboratory personnel during and following the assessment. Everyone has been courteous and helpful, and all personnel interviewed were clearly highly competent, poised, and articulate about their tasks. If you have any questions, please let me know. You may contact me at 802-233-2473 or by email at dgaynor@phoenixchemistryservices.com.

Sincerely,

Deborah H. Gaynor

Digitally signed by Deborah H. Gaynor
DN: cn=Deborah H. Gaynor, o=Phoenix Chemistry Services, ou, email=dgaynor@phoenixchemistryservices.com, c=US
Date: 2016.07.23 07:06:40 -04'00'

Deborah H. Gaynor, Ph.D.

cc: Rhonda Kay
Amy McCormick

Technical Project Audit: TestAmerica, Inc.-Canton, June 1-2, 2016

Audit Reference #: 2016-0602

Section 1: Introduction

This is the final findings report for a technical project audit performed at the TestAmerica, Inc.-Canton (Canton) facility located at 4101 Shuffel Street NW in North Canton, OH on June 1 and 2, 2016. The audit was performed at the request of the Project Manager for the Engineering Evaluation/Cost Analysis (EE/CA) being performed at the former Jaite Paper Mill (Jaite) at Cuyahoga Valley National Park in Sagamore Hills, Ohio. The Johnson Company of Montpelier, VT has been retained by the National Park Service to prepare the EE/CA planning documents for the Jaite project. The Canton laboratory is the primary laboratory, and it's subcontracting laboratories TestAmerica-Pittsburgh and TestAmerica-Knoxville will perform specific tests. Laboratory services for asbestos analysis are outside the scope of this audit.

Deborah H. Gaynor, Ph.D., representing Phoenix Chemistry Services of N. Ferrisburg, VT performed the audit. The purpose of the audit was to verify conformance with project requirements as specified in the Quality Assurance Project Plan (QAPP; Draft, May 20, 2016, Appendix 2 of the Sampling and Analysis Plan), and to determine whether sub-contracted analyses will be prepared by the Canton laboratory and processed by the sub-contracting laboratories according to project objectives and in accordance with guidance for incremental sampling methodology (ISM). The audit included the opportunity for all personnel involved with receipt, handling, analysis, and reporting of project samples to convey any concerns with inter-laboratory communications and systems, and allowed the auditor to observe laboratory protocols in practice.

The scope of the audit included sample receiving and storage, sample preparation and analysis for the organic, inorganic, and general chemistry methods, including in particular the ISM sample processing area and procedures, data reporting, quality assurance, and project management sections of the Canton laboratory. Documents reviewed during the audit included the QAPP; method sources; laboratory SOPs, current control charts, and current method detection limit study summaries (MDLs) for all methods; and laboratory logbooks, checklists, and electronic tracking systems for sample receiving, log-in, handling, instrument analysis and instrument maintenance. Full citations for published documents reviewed during the audit are presented in Appendix A.

Section 2: Personnel

Contact information for parties involved in the site project management and sampling; laboratory project management, sample handling, analysis, and quality assurance is given in Appendix B. Personnel interviewed during the audit, and their roles in the laboratory, are indicated. Contact information for the site owner and the respondents, and/or their designated representatives, are provided in the QAPP.

The audit began with an opening meeting attended by Carolynne Roach, the Laboratory Director; Ray Risden, the laboratory Technical Director; Aaron Martin, the Operations Manager; and all Quality Assurance and Department Managers listed in Appendix B (or lead analysts if the Manager was unavailable). The audit closing meeting was attended by Carolynne Roach, Mark Loeb, Melissa Fuller-Gustavel, and most of the Department Managers who had been interviewed during the audit.

Section 3: Summary of Findings

Volatiles

At the laboratory's request, the audit started in the Volatiles Department in order to prevent possible cross-contamination from the sample receiving area. Volatiles Department Supervisor, Tom Stiller, was interviewed; the laboratory has an 8-person analytical staff on various shifts, covering approximately a 12-hour work day for 6 days a week. Project samples will be analyzed by Method 8260C; as presented in the SOP, the laboratory uses a minimum six point calibration curve. Template instructions for making calibration standards are readily available in the TALS system, and traceability is instantly accessed through barcodes on all reagent and standards containers and barcode readers next to each terminal. As demonstrated through examination of a current calibration on a randomly chosen instrument, most analytes are performing well, with average response factor calibrations. Tom noted that bromoform is the most frequently problematic analyte.

The auditor asked how methanol samples are handled, since the surface soils will be collected as 200 gram (g) ISM samples. The containers are 500 milliliters (mL) with 200 mL methanol; the lot number is tracked with a barcode assigned to the lot, which typically consists of numerous cases of 1 L bottles. The methanol is checked for cleanliness at the corporate level prior to release of the lot to all network laboratories. One bottle is placed in the refrigerator when containers are sent out into the field for use in making method blanks and laboratory control samples; this is a commendable corporate policy which is in accordance with method requirements, but which is not followed by many otherwise reputable laboratories.

Methanol-preserved samples are analyzed with both an aqueous "window" blank and a methanol method (preparation) blank. The laboratory controls all analytes in blanks to less than the reporting limit (aqueous), except methylene chloride and acetone, which are allowed to be present in blanks at concentrations up to three times the aqueous reporting limit. Autosamplers are used to add the surrogates and internal standards. All volatile samples are screened on "retired" instruments prior to analysis.

The Volatiles Department has five instruments and three analysts for aqueous samples; three instruments and two analysts for low-level soil samples; and two instruments and one analyst for TCLP samples. One analyst prepares low and medium level soil samples. Autosamplers are used with each instrument, and are typically kept in designated pairs; most instrument pairs are an Agilent GC/MS with OI autosamplers, and a #10 OI trap is used. Data acquisition is performed by HP Chemstation, and data integration and quantitation is performed by the TestAmerica proprietary Chrom software.

The reporting of dilutions was discussed; the upper half of the calibration range, based on one-half of the high standard of the calibration curve, is targeted for dilution of the most concentrated analyte. Tom noted that if we wish to request that a higher concentration analysis be performed and reported, we may request "multiple dilutions for the lowest reporting limits".

Sample Management

The auditor was given a general tour of the Sample Receiving area by the Sample Receiving Manager, Ann Maddux. Ann introduced the laboratory use of the "backlog report" which is how samples and their holding times and reporting dates are tracked within the proprietary laboratory information management system, known within the TestAmerica laboratories network as TALS (TestAmerica Laboratory information System). Coolers are unpacked, priority analyses (short holding times) are noted and cued for immediate log-in, and remaining samples are organized according to appropriate handling (volatiles are logged in first, or are put on carts to be held in the walk-in cooler until they can be logged in). Volatiles are ultimately moved to a separate walk-in cooler for storage following log-in. The main walk in cooler is actually quite a large area with several rooms, and contains extensive shelving to organize samples. Smaller coolers or room-temperature cupboards are used to store archive samples following analysis and prior to disposal.

Any discrepancies noted during sample receipt and log-in are brought immediately to the project manager's attention for resolution; photographs of illegible labels or compromised containers may be taken, and become part of the permanent record. When asked, Ann said that samples are only noted as containing ice when the entire container is frozen, as this may break or leak when it thaws. The auditor noted that many data validators rely on the laboratory to report when ice is present in a sample container, as this may indicate that the sample had frozen on the way to the laboratory, and was in the process of thawing out again. This was discussed again during the close out meeting (see discussion below).

Analysts are given the shelf number on their "backlog reports" to aid in locating their own samples when they are ready to start sample preparation. Samples are assigned laboratory numbers and a letter designated the container; both the laboratory identifier and the container identifier are shown on the "backlog report", so analysts match both when selecting containers for sample preparation.

The shipping area is adjacent to the sample receiving area. Samples which will be sent to the sub-contracting laboratories will be returned to the shipping area for packing and shipping following ISM processing.

General Chemistry Department

Analysts for the Walkley-Black total organic carbon (TOC) method include Julie Sanford and Tom Harshman (both interviewed) Analysts for pH, hexavalent chromium, and hardness include Diem Nguyen, Gabrielle Renner, and Jill Burns (all interviewed); the Department Manager, Lucas Grossman, was out of the laboratory during the audit. All analysts were experienced, able to clearly demonstrate or talk through their test(s), and were comfortable answering questions and discussing details of the analytical procedures. Several minor findings were noted in this area, and are discussed below.

The General Chemistry department uses printed copies of SOPs (because there are so many of them), and these were quickly accessed during the audit in response to a question, and are clearly relied upon by the analysts. As in all other laboratory departments, General Chemistry uses electronic records rather than printed bench sheets. However, one analyst described using paper to record buret readings, which she then typed into the electronic bench sheet, after which she discarded the paper; the Quality Assurance Manager stayed behind when the auditor moved on to the next interview to correct this behavior, and to remind the analyst that laboratory policy requires that any written record must be scanned into the permanent record. The auditor also noted that the hexavalent chromium calibration summary report (not the final CLP-type form) does not include identification of the analyst, the instrument, or the date, which can increase the complexity of data validation; only the batch identifier provides a cross-reference to the other identifications. Also in General Chemistry, some analysts indicated that access to a terminal was sometimes competitive, and that seemed to be a minor issue.

Excel spreadsheets are used to record pH, titrimetric, and spectrophotometric results. No electronic connection exists for either the pH meter or the spectrophotometers, so the analyst must manually transcribe the buret or spectrophotometer readings to these spreadsheets. Terminals are placed close to the instruments, although in some instances the analyst records the readings on a printed sample log prior to typing them into the spreadsheet. With a single analyst/method exception, these written records are scanned into the permanent record, and reported when a full data package is submitted. Transcription errors are a vulnerability whenever a direct electronic record is not taken, and the laboratory acknowledged that a direct electronic connection is on their wish list. The auditor also noted that at least one of the Excel spreadsheets did not have cell protection for the cells containing formulas. One laboratory manager (Diane Jones) has been responsible for implementing cell protection and other spreadsheet security measures (the spreadsheets become locked 24 hours after analysis, allowing time for data review corrections, but preventing any later substitutions in the spreadsheets). While these security measures are reviewed annually, the loss of the formula cell protections was a surprise, and laboratory management discussed implementing immediate corrective action for this situation during the audit close out meeting.

Laboratory personnel asked whether the Walkley-Black or Lloyd Kahn method will be used for TOC; the QAPP references Walkley-Black, but the quote references only Lloyd Kahn, which is performed at Pittsburgh, but not at Canton. The auditor stated that Walkley-Black is the selected method for sediment samples.

ISM Sample Processing

Diane Jones is the lead analyst for the ISM sample processing laboratory, and was a member of the original ITRC team which wrote the guidance documents for this methodology. Diane gave a tour of the ISM processing laboratory, which is a room with a dedicated air handling system, a drill press, a hydraulic press, two crushers, a disaggregator, a sieve, and a ball mill. The selected method of sub-sampling is the two-dimensional slab cake method, although a one-dimensional slab cake is also occasionally used as needed. A large (walk-in) drying oven which maintains a constant warm temperature and good (filtered) air flow across the slab cake trays is located within the room.

Diane used a sample from another project to demonstrate the layout of the slab cake and the scooping method used to create the individual samples for the different tests. We discussed the

sample mass requirements for all the tests, and identified that the AVS/SEM test to be performed in Pittsburgh, and the PCB congeners test to be performed in the Knoxville laboratory do not have sample mass requirements that have been communicated to Canton. Diane contacted both laboratories during the audit, and will determine what Pittsburgh will need; she was told that the congeners will require a 1 g sample for screening, and a 10-12 g sample for analysis. She will plan to make a smaller sample at the same time in case a dilution is necessary (because this is an isotope dilution analysis, dilutions must be re-extracted). Under the current plan, these processed soil samples will be held, frozen at -10ºC, until all other results are reported. An additional back- up aliquot at 10-12 g may also be set aside at this time, at the discretion of the two laboratories working together to determine the best storage option.

We discussed the concrete samples, and Diane noted that she will take these down to the maximum 2 mm particle size, but due to the nature of concrete, she cannot put these on the ball mill and reduce the size any further. Any larger particles of rock used in the formation of the concrete will be excluded from the final sample during the sieving process. Similarly, samples containing ballast or rocks from the old rail bed will not be crushed so as to exclude discrete rocks larger than ½ to 1 inch in size.

Diane noted that silty or sandy samples which are high in PCBs or samples that contain moderate to large amounts of vegetative material will tend to form "nuggets" during processing. These nuggets may be reduced to less than 2 mm particle size by the use of the ball mill, although this adds significant time and cost to the processing. Diane noted that using the ball mill tends to produce samples with similar or perhaps slightly higher concentrations, but with much tighter confidence limits. Later discussion with Rhonda Kay indicated that oily samples or samples with significant vegetative material are unlikely to be submitted, but she will keep this in mind during field sampling.

We discussed the request for a laboratory duplicate; Diane will select which sample will be used for this, and will include one for every parameter.

Other questions which the laboratory needs instruction on include the following: Diane asked whether a percent moisture analysis is needed on the dried ISM samples. The laboratory's experience is that the final moisture content is about 1-2% when the sample has reached the "crumbly" end point. Diane also asked whether it is correct that hardness analysis has been requested in the soil samples, or if this should be determined by calculation from the metals analysis. To analyze soil samples for hardness, an 18-hour leach must be performed, followed by filtration (.045 micron) to create a liquid sample for titrimetric analysis. Diane asked how many equipment rinse blanks were required, and at what times during the project, and for which tests. The auditor pointed out that the QAPP (Worksheet #11) specifies that one equipment rinse blank should be performed for shared equipment for every 20 decision units, or discrete samples, or per media. Diane noted that this will be incorporated into the project protocols. We also discussed that (as specified in Worksheet #11 of the QAPP) the samples for pH analysis will be taken from the moist slab cake at the same time as the samples for volatiles percent moisture, since the effect of drying on pH analysis is unknown.

During the closeout, it was discussed that the hand-written final sample weight that Diane records for the ISM sub-samples is used by the organics extraction analysts, but this is not scanned and kept as a paper record, because she thought they were re-weighing the samples.

The auditor noted that while hearing protection gear was present in the ISM laboratory, neither a face shield or a respirator was present. Diane stated that the face shield is shared with facilities personnel, and was probably in use in another equipment room. She will talk to Steve Jackson, the environmental health and safety coordinator for the laboratory, about the use of a respirator while processing samples which might contain low levels of asbestos.

Inorganic Methods (Preparation and Analysis)

Sample preparation analysts for metals include Kyle Dillon and Alex Colosi (both interviewed), instrumental analysts include Roger Toth and the Department Supervisor Karen Counts (both interviewed), and Kyle for mercury analyses. Kyle eloquently presented, with support from Alex, how soil and water samples are digested for metals analyses, and how mercury samples and calibration curves are digested and analyzed.

We discussed in detail how the ISM samples are handled during sample digestion. ISM solid sample digestion for both the ICP methods (10 g ISM sample each) and mercury (3 g ISM sample) is performed in five containers that allow appropriate volumes for the hot-block digesters, following which the sub-sample digestates are combined. The standard (non-ISM) sample size for mercury analysis is 0.6 g, which allows for easy division ($1/5^{th}$) of the reagents and spikes used.

Roger is the lead ICP/MS analyst, and Karen is the lead ICP/AES (also referred to as ICP/OES) analyst, though both may perform analyses on both instrument types. Both participated in the discussion of these two analytical methods. All discrete soil analyses, and the soil MDLs, are based on a 1:2 dilution of the digestate; aqueous samples are analyzed without dilution, and the aqueous MDLs reflect this. Separate MDL studies were deemed unnecessary for ISM soils, since these are digested at the same volume of reagents and spikes as the discrete samples (and the laboratory uses both a 1 g and a 2 g sample mass in its routine digestion); the digestates are combined after digestion, but the sample volume analyzed is also the same as for a routine discrete soil sample. Like discrete soil samples, discrete sediment samples are analyzed at the same 1:2 dilution of the digestate, so sediment detection limits correspond to the submitted MDLs. However, and of particular note, is that for ISM solid samples (soils and sediments), the digestate is further diluted 5-fold for ICP/MS (Method 6020) analysis but analyzed full strength on the ICP/AES instrument. This dilution causes the relative detection limits for barium, beryllium, chromium, copper, lead, nickel, thallium, and vanadium to be higher in the ICP/MS analysis than in the ICP/AES analysis. The Johnson Company has the option of requesting analysis by "6010/6020", leaving the laboratory to select the best analysis on a per-element basis, which is likely the best choice to ensure the lowest detection limits.

Otherwise, the two instruments both provide reliable analyses for our list of analytes; boron may be problematic on the ICP/MS, but this is not a project analyte. Roger and Karen also noted that due to matrix interferences with the large sample mass in ISM soils, dilutions as large as 50-fold may be encountered for the ICP/MS analysis (we did not discuss potential maximum dilutions on the ICP/AES). Sometimes this is due to a true inter-element interference, and sometimes it is due to suppression of the internal standard, but it may not be possible to distinguish the reason for large dilutions from the data package (the Narrative will simply say "diluted due to matrix"). If the validator wishes to follow up on the reasons for dilutions, the laboratory may be asked for

clarification, and the analyst will review the data and comment on why the dilution was performed.

Karen and Roger also briefly discussed the instrumental capabilities of the laboratory. ICP/MS analyses are performed on an Agilent 7700 with an Agilent/Cetac autosampler (ASX-500) ; a second ICP/MS instrument (an X series 2 with a collision cell) is not currently in service. The working instrument is outfitted with high matrix interference technology, that doubles the gas flow through the analyzer, reducing the sample mass introduced to the cones. This technology is used 100% of the time, and the MDL studies are performed with it on. All standards, blanks, quality control, and field samples are analyzed using this technology. The ICP/AES analyses are performed on a Thermo ICAP I9 and an I12 with similar autosamplers.

Organic Methods (Preparation and Analysis)

Sample preparation analysts for extractable methods (semivolatiles, pesticides, PCBs, and herbicides) include the Department Supervisor Chris Coast (Extractions, interviewed), and the technical leads Justin Ross and Caitlin Scott (both interviewed). Aqueous semivolatile extractions may be performed using either separatory funnel or continuous liquid-liquid extraction (CLLE); currently, only separatory funnel extraction is included in the QAPP. Justin walked through both extractions demonstrating laboratory capacity (there are a significant number of units available for both techniques), and discussing the applicability of the two techniques to the analytical tests.

The laboratory strongly recommends that CLLE should be used for pesticides and PCBs analyses, and that separatory funnel produces the best results for semivolatiles. In addition to the routine protocols, a reduced volume extraction using one 250 mL volume of aqueous sample, may be performed for semivolatiles (and also for TPH diesel analysis, which is not needed for this project); this will be covered in more detail in the instrumental discussion below. Justin then walked through extraction procedures for solid samples. The laboratory provides both sonication, which is included in the QAPP, as well as Soxhlet extractions. The laboratory recommends that Soxhlet extraction provides the best results for semivolatiles, pesticides, and PCBs. Capacity may be an issue as well, since the laboratory has almost 170 set-ups for Soxhlet extraction but only two sonicators. We discussed that many of the soils for this project would be processed first by ISM, and the extraction analysts were both familiar with the sample weight requirements, and the necessity to use the entire sample mass provided. The analysts also noted that samples processed by ISM should not be sonicated, since this may produce a gel.

We discussed in particular that the laboratory processes pesticides and PCBs as separate samples, bringing the pesticide extract to a final volume of 5 mL and the PCB extract to a final volume of 2 mL. The procedure can be modified to produce a single final extract, but this must be specified prior to sample receipt. This means that separate laboratory ISM sub-samples must be produced from each field ISM sample, but this is a standard procedure for the laboratory.

In the instrumental sections, the semivolatiles (SVOCs) Department Supervisor Tom Hula and gas chromatography (GC) Department Supervisor Olguita Colon were both interviewed, along with PCB lead analyst Heather Bosworth.

The SVOC department has 4 Agilent GC/MS instruments and three analysts. All instruments are equipped with Agilent autosamplers. In addition, there is a newer Agilent GC/MS instrument

which has the capability to run large volume injections (5 uL instead of 0.5 uL). This capability is used for the reduced volume extractions, and the combination achieves the same reporting limits as obtained for the standard 1 L sample volume. All samples are screened prior to analysis (at 1:20 following the end of the 12 hour analytical window on any instrument), and as in the VOCs department, Tom Hula noted that if dilutions are needed, only the acceptable analysis is reported unless the request for "multiple runs for the lowest reporting limits" is made to the project manager prior to sample receipt. We discussed calibration procedures and corrective actions for various analytical exceedances; all responses were consistent with laboratory SOPs and good laboratory practices. Tom noted that the only common laboratory contaminant that poses recurring problems is (bis)2-ethylhexyl phthalate, which typically is found at about 2 parts per billion (ppb); since this is a project analyte, low-level blank contamination may be an issue.

Heather demonstrated the typical chromatography for the PCB Aroclors analysis, and both she and Olguita discussed the similarities and differences in analysis of pesticides and PCB Aroclors. Both methods use the internal standard modification, and have similar calibrations and data analysis procedures for selecting the peaks to be used for the multi-component analytes. The laboratory includes full five point calibrations for chlordane and toxaphene (the two multi- component analytes in the pesticides method), and for the PCBs, the calibration is analyzed using pairs of Aroclors that exhibit little to no overlap in peak distribution. Three to five peaks are used for the PCB Aroclors, five peaks are used for toxaphene, and 4 peaks for chlordane. Area, not height, is used for quantitation. The analysts perform cleanup for sulfur in sample extracts as a routine procedure, but additional cleanup procedures for other interferences for the PCB Aroclors (silica gel and florisil or alumina column) must be requested prior to sample receipt.

The laboratory reports each analyte from the best column (all instruments are equipped with two columns and two detectors); an individual sample result might include results from both columns. The analyst's judgment is used to select the column to report as the primary result; if the analyst judges that interference is present on one column, the lower result will be reported. There are two GC instruments used for pesticides, with one primary analyst and one backup analyst; five instruments for PCBs, with three primary analysts and one shared backup analyst, and one instrument for herbicides, with one primary analyst (who was not present on the day of the audit), and the Department Supervisor as backup analyst. Two instruments are available for screening, and all samples are screened.

Glassware cleaning procedures, reagent and standards traceability, and the use of the "backlog report" for organizing sample priorities were discussed in all departments. Terminals and barcode scanners were present throughout the extraction and analytical laboratories, and all comments and procedures were consistent with other departments.

Data Handling and Review

During the audit, each analytical section demonstrated the in-laboratory data handling systems within TALS (the laboratory information management system). All network laboratories use the single system for reporting; different functionalities are available and may be requested to customize a set of reports. The TestAmerica laboratories use both internal and external data reviewers. The primary analyst performs primary review, checking all barcoded entries and the worklist, and then either another department analyst or the Supervisor, or an external data reviewer, performs full secondary review, checking calculations, dilutions, and a general "reasonableness" check, with follow-up as indicated if issues are noted.

External data review is performed for the Canton laboratory by a former senior analyst now living in Chicago who reviews the General Chemistry tests, and by an experienced analyst in a network facility in Thailand who reviews data for the other laboratory sections. This provides the advantage of overnight data review, while the analysts and supervisors are not at work, which keeps data flowing more smoothly through the laboratory.

While there are very few hand-written records produced, the auditor requested that scans of any hand-written records be included in the final data packages as a deliverable.

Project Management

The Project Manager (PM) for the Jaite project at Canton is Amy McCormick. As PM, Amy is responsible for coordination of sample containers shipping to the field, sample receiving and scheduling into the laboratory, resolution of sample receiving discrepancies, coordination with the designated sub-contracting laboratories, client communications, and data package reporting. She oversees the arrival of samples; handles any questions which arise during log-in or analysis; specifies report formats and ensures completeness and conformance with client specifications; writes the Case Narrative; and posts reports on the laboratory's client file-server system. Specific requests, such as the addition of project-specific control limits, which would enhance the data assessment (data validation) process, are implemented by the PM.

If a problem is identified at the time of sample receipt, it is referred to the PM for resolution.

Quality Assurance Systems

While the technical performance of a laboratory is dependent on appropriate and thorough Quality Assurance (QA) management, this was not a major focus of the audit. The QA Manager, Mark Loeb, was present for both the opening and closing meetings, and accompanied the auditor for both days, and the other two members of the QA staff, Dorothy Leeson and Melissa (Mel) Fuller-Gustavel, were also present for the bulk of the audit. Dorothy was out of the laboratory on previously planned travel on June 2, but spent time going over control charts with the auditor on June 1. Mark and Mel noted quality systems issues during laboratory interviews and were already addressing the few issues identified in the audit prior to the close of the audit.

It was mentioned during the audit that the laboratory has the ability to implement project-specific control limits for all requested parameters. This will make validation much easier. The auditor notes that this does not impose additional restrictions or requirements on laboratory performance of these methods; it is implemented at the reporting stage in order to simplify the data review and assessment phase.

All records other than maintenance activities are kept electronically. Each laboratory (except the ISM processing room) has at least one terminal present, and all SOPs, checklists, work orders (referred to as the "backlog report"), and results records are maintained electronically and are available to all analysts at all times.

All laboratory departments are using fairly new equipment maintenance logbooks printed and bound by Mel, which contain a section for routine maintenance, and a section for more intensive repairs or maintenance activities. The logbooks are paginated, were in use and completed appropriately with analyst initials and dates as needed. The analysts seem to like these logbooks and compliance with their use appears strong.

The Canton laboratory maintains NELAP accreditation for all methods, analytes, and matrices listed in the QAPP, through one or more of the accrediting authorities in Illinois, Florida, Kansas, Minnesota, New Jersey, New York, Oregon, Pennsylvania, Texas, and Virginia, and maintains state certifications through one or more of the following state programs: Connecticut, California, Kentucky, Washington, West Virginia, Wisconsin, and the Ohio VAP.

In particular, the Ohio VAP certificate includes most of the methods and matrices listed in the QAPP, including the Walkley Black method, which is also NELAP-accredited through Texas, Oregon, Minnesota, New Jersey, and Pennsylvania.

Department of Defense (DoD) certification has recently been dropped by the laboratory, and they are in the process of updating SOPs to remove references to DoD Quality Systems Manual (QSM) requirements; some SOPs referenced in the QAPP still contained these references at the time of the audit; these should be disregarded if any remaining SOPs are not replaced prior to the field season.

Section 4: Proficiency Testing Results

The auditor requested copies of the laboratory's NELAP-required proficiency testing (PT) study results for the last two testing rounds, and Dorothy Leeson submitted results from three pairs of aqueous and solid studies starting in early 2015 and continuing through the spring of 2016, as well as the results of five additional (rapid response, also called "make-up") studies for specific analytes which had been unacceptable in the routine studies. These results were examined following the audit, and while acceptable performance was exhibited for most analytes, in both solid waste and "water pollution" (i.e., non-potable water) matrices for the methods to be used in this project, the following deficiencies for project analytes by project-selected methods were noted:

Matrix	Analyte	Analytical Method	Units	Assigned Value	Result	Acceptance Range
Phenova Study HW0115; closing date: March 19, 2015						
Soil	Silver	TCLP-6010B	mg/L	7.03	13.2	3.35 - 10.7
Phenova Study WP1015; closing date: November 19, 2015						
Water	Dieldrin	8081A	ug/L	2.22	1.04	1.10 - 3.02
Water	Endrin	8081A	ug/L	5.87	2.24	2.57 - 8.35
Phenova Study HW0116; closing date: March 10, 2016						
Soil	Benzene	8260C	ug/Kg	66.4	126	38.8 - 91.9

Soil	Dichlorobromomethane	8260C	ug/Kg	119	246	77.4 - 164
Soil	Bromoform	8260C	ug/Kg	132	308	66.9 - 196
Soil	2-Butanone	8260C	ug/Kg	475	916	159 - 728
Soil	Carbon tetrachloride	8260C	ug/Kg	30.3	55.6	14.9 - 45.1
Soil	Chlorodibromomethane	8260C	ug/Kg	86.0	194	51.5 - 119
Soil	Chloroform	8260C	ug/Kg	72.7	145	43.6 - 102
Soil	1,2-Dibromo-3-Chloropropane	8260C	ug/Kg	196	489	128 - 278
Soil	1,2-Dichlorobenzene	8260C	ug/Kg	92.6	180	44.4 - 131
Soil	1,3-Dichlorobenzene	8260C	ug/Kg	28.0	52.0	8.38 - 41.5
Soil	1,4-Dichlorobenzene	8260C	ug/Kg	162	300	55.7 - 229
Soil	1,1-Dichloroethane	8260C	ug/Kg	164	312	93.4 - 236
Soil	1,2-Dichloroethane	8260C	ug/Kg	77.8	140	45.5 - 109
Soil	1,1-Dichloroethene	8260C	ug/Kg	83.8	141	40.5 - 130
Soil	trans-1,2-Dichloroethene	8260C	ug/Kg	124	230	78.1 - 177
Soil	1,2-Dichloropropane	8260C	ug/Kg	76.0	153	44.7 - 103
Soil	Ethylbenzene	8260C	ug/Kg	125	244	69.7 - 179
Soil	2-Hexanone	8260C	ug/Kg	239	560	99.2 - 354
Soil	Methylene chloride	8260C	ug/Kg	36.0	59.2	16.5 – 55.3
Soil	Methyl-tert-butyl ether	8260C	ug/Kg	162	331	68.3 - 237
Soil	Tetrachloroethene	8260C	ug/Kg	152	295	68.3 - 219
Soil	Toluene	8260C	ug/Kg	40.0	75.6	22.4 – 56.7
Soil	1,2,4-Trichlorobenzene	8260C	ug/Kg	128	243	51.3 - 205
Soil	Trichloroethene	8260C	ug/Kg	189	374	97.4 - 269
Soil	Nitrobenzene	TCLP-8270D	ug/L	268	369	67.6 - 349
Soil	Mercury	TCLP-7470A	ug/L	2.84	3.58	1.13 - 3.23
Phenova Study WP0416; closing date: May 19, 2016						
Water	Vanadium	6020A	ug/L	63.2	32.3	53.7 - 72.7

Although rapid response studies (which are taken to re-establish the laboratory's acceptable performance for an unacceptable score on a recent PT study) were submitted, these studies did not correspond with the analytes with unacceptable scores. Acceptable performance on rapid response studies immediately following unacceptable routine PT studies should be submitted to demonstrate the laboratory's capability to accurately analyze site samples.

Section 5: Health and Safety Findings

The Environmental Health and Safety Coordinator (EHSC), Steve Jackson, is shared with the TestAmerica-Pittsburgh laboratory; Steve was out of the laboratory during the audit. Few safety concerns were noted during the two day audit. Overall, the laboratory appears to maintain an acceptable health and safety program, including general laboratory operations, training programs, and staff compliance. The only safety concern noted was that the ISM Technical Lead, Diane Jones, expressed concerns about potential asbestos content in the dust generated during ISM processing, for the samples for which asbestos testing has been indicated. She will discuss these concerns with Steve Jackson, and formulate a plan for dust management. Highlights of the overall laboratory health and safety systems include the following findings.

- All personnel receive comprehensive health and safety training on an on-going basis

through personal training.

- Different colored lab coats are worn by visitors and actively working laboratory personnel, and designated areas are marked where laboratory coats are removed and these areas are "safe zones" for light snacks. Inside the laboratory work areas, keyboards are marked either as "gloves only" or "no gloves" to prevent contamination through keyboard surfaces.
- Appropriate personal protective equipment is widely available and worn consistently throughout the laboratory. It was noted that no respirator was present in the ISM processing laboratory, and the Technical Lead noted that a large amount of dust is generated during sample processing. Hearing protection was present in the room, but Diane stated that the face shield appeared to have been taken to another room for use and this was not seen during the audit.

List of findings:

- One analyst described using paper to record buret readings, which she then typed into the electronic bench sheet, after which she discarded the paper.
- During sample check-in, Sample Receiving staff only note if completely frozen containers are received, as these may break or leak when they thaw.
- At least one of the Excel spreadsheets used in General Chemistry did not have cell protection for the cells containing formulas.
- The QAPP states that no target analytes should be found in laboratory blanks at concentrations above the reporting limit, but laboratory managers and analysts reported that methylene chloride and acetone, are allowed to be present in blanks at concentrations up to three times the aqueous reporting limit, and bis(2-ethylhexyl)phthalate is typically found in semivolatile method blanks below the aqueous reporting limit at approximately 2 ppb.
- A single dilution analysis is usually reported for samples for which one or more analyte exceeds the calibration range.
- Although laboratory policy requires that any written record must be scanned into the permanent record, one analyst described making notes of buret readings on scrap paper before transcribing these to the laboratory information management system (TALS).
- The instrumental calibration report for hexavalent chromium calibrations does not include identification of the analyst, the instrument, or the date, although the batch is present on the page. This increases the complexity of data validation.
- In the General Chemistry Department, some analysts noted that a terminal was not always readily available for data input, so tests had to be scheduled amongst the analysts in order to ensure immediate access for data input.
- The auditor noted that at least one of the Excel spreadsheets in use for General Chemistry did not have cell protection enabled for one or more cells containing formulas.
- Laboratory personnel asked whether the Walkley-Black or Lloyd Kahn method will be used for TOC; the QAPP references Walkley-Black, but the quote references only Lloyd Kahn, which is performed at Pittsburgh, but not at Canton.
- Diane will determine what sample mass Pittsburgh will need for the AVS/SEM analyses, and will ensure that the analysts there understand how to properly handle and analyze ISM samples.
- Although described in the ITRC guidance as an option, a laboratory duplicate for ISM processing is not routinely performed.
- Diane asked whether a percent moisture analysis is requested for the dried ISM samples.

- The QAPP currently implies that hardness analysis has been requested in the soil samples, rather than determination by calculation from the metals analysis.
- Diane asked how many equipment rinse blanks were required for ISM processing equipment, and at what times during the project, and for which tests.
- Diane asked if the ISM samples for pH for the volatiles fraction could be sub-sampled prior to drying, since the impact of drying on pH is unknown.
- During the closeout, it was discussed that the hand-written final sample weight that Diane records for the ISM sub-samples is used by the organics extraction analysts, but this is not scanned and kept as a paper record, because she thought they were re-weighing the samples.
- The metals digestate is diluted 5-fold for ICP/MS (Method 6020) analysis but analyzed full strength on the ICP/AES instrument.
- Separate laboratory ISM sub-samples are produced from each field ISM sample for pesticides and PCB Aroclors analysis, since these are routinely treated independently by the extraction procedures.
- If additional cleanup procedures are desired for interferences (other than sulfur) for the PCB Aroclors (silica gel and florisil or alumina column), these options must be requested prior to sample receipt.
- Some laboratory SOPs still contain references to DoD Quality Systems Manual (QSM) requirements; since the laboratory is no longer participating in this program, the SOPs are being updated to remove these references.
- The laboratory has the ability to implement project-specific control limits for all requested parameters.
- The laboratory strongly recommends that continuous liquid-liquid extraction (CLLE) should be used for aqueous pesticides and PCBs analyses, and that separatory funnel produces the best results for aqueous semivolatiles, and that Soxhlet extraction provides the best results for semivolatiles, pesticides, and PCBs in solid samples. It was also noted that samples processed by ISM should not be sonicated, since this may produce a gel.

Section 5: Conclusions, Recommendations, and Suggested Corrective Actions

Each of the findings in this section has been numbered, and is followed by the laboratory's response and/or corrective action, or the changes made to the QAPP or other planning document, along with any comments deemed necessary by the auditor regarding acceptability of the response, or if further clarification or action was deemed appropriate. Findings without the Response and Evaluation comment are maintained as observations not requiring any action.

The laboratory has requested a kick-off meeting for laboratory and field personnel and the validator, and the auditor concurred with this request. Agreements made during this meeting will be recorded in a Field-Laboratory Coordination Memorandum, which will summarize the details discussed, and provide a record of any details not captured in the QAPP. These items, along with other actions which remain pending are highlighted below with **boldface** font.

List of conclusions:

1. At the closing, the auditor discussed the issue of whether or not the presence of ice should be noted during sample check-in. The auditor stated that many other laboratories will state if a liquid sample contains any ice, and that validators rely on these statements for judging sample integrity.

Response and Evaluation: no response received; no action required.

2. The TestAmerica policy and procedures for methanol-preserved volatiles samples is commendable.

Response and Evaluation: no response received; no action required.

3. Project action limits in sediments for acetone and methylene chloride are below laboratory reporting limits without the presence of contamination, and for bis(2-ethylhexyl)phthalate are at or below laboratory reporting limits for surface water and porewater. The laboratory should be aware that samples may be qualified for method blank contamination, and results will be evaluated for completeness based on the achievement of project action limits.

Response and Evaluation: no response received; no action required.

4. The routine 5-fold dilution of the metals digestate causes the relative detection limits for barium, beryllium, chromium, copper, lead, nickel, thallium, and vanadium to be higher in the ICP/MS analysis than in the ICP/AES analysis.

Response and Evaluation: A footnote will be added to Worksheet #15 in the QAPP noting that the metals analysis should be by "6010/6020 for lowest reporting limits". Acceptable; **this information should be reviewed in a kick-off meeting, and should be captured in the laboratory Project Notes and in a coordination document**.

5. For the metals analyses, the Case Narrative will simply say "diluted due to matrix" if a dilution was needed. If the validator wishes to follow up on the reasons for dilutions, the laboratory may be asked for clarification, and the analyst will review the data and comment on why the dilution was performed.

Response and Evaluation: **The validator should be aware that this option is available**.

6. The project manager needs further instruction regarding what size containers to provide for porewater samples, and how to prioritize analyses if limited sample volume is obtained.

Response and Evaluation: Worksheet #19 has been edited to show that two 250 mL bottles will be needed for porewater samples. Acceptable.

List of suggested corrective actions:

7. The Quality Assurance Manager immediately correct the analyst who described discarding a paper record, and instructed the analyst that any written record is required to be scanned into the permanent record. Documentation of this re-training should be added to the analyst's personnel records, and a statement that this has been done (without reference to analyst identity) should be included in the audit response. The auditor requested that any manually recorded information should be included in the final data package, as well as maintained in the electronic project archives.

Response and Evaluation: telephone response received; lack of documentation is tolerable.

8. The hexavalent chromium calibration summary report page should be annotated with additional cross-reference information to improve transparency and traceability.

Response and Evaluation: telephone response received; **validator will determine if corrective action is sufficient**. If not, this audit will be cited in validation report.

9. All Excel spreadsheets in use should be immediately reviewed to ascertain the status of

the formula cell protections, and corrected if the cell protections have been lost. A more formal and more frequent review schedule should be implemented to ensure that errors do not creep into these software tools.

Response and Evaluation: telephone response received; acceptable.

10. The Johnson Company has the option of requesting analysis of the ISM solid samples by "6010/6020", leaving the laboratory to select the best analysis on a per-element basis, which is likely the best choice to ensure the lowest detection limits. This option should be requested and incorporated into the project quote.

Response and Evaluation: telephone response received; acceptable. **COC should read "6010/6020 for lowest RLs", and this should also be incorporated into laboratory Project Notes and a coordination document.**

11. The appropriate rapid response studies analyzed immediately following unacceptable routine PT studies should be submitted to demonstrate the laboratory's capability to accurately analyze site samples.

Response and Evaluation: telephone response received and laboratory asserted that all rapid-response PT studies were successfully analyzed and reported, and received acceptable scores. **Study results must be submitted**.

12. The QAPP and the quote should be modified to include continuous liquid-liquid extraction (CLLE) for aqueous pesticides, and Soxhlet extraction for semivolatiles, pesticides, and PCBs in solids.

Response and Evaluation: the QAPP has been modified accordingly; acceptable. **A new quote has not been issued, but must reflect the appropriate extraction methods; pending.**

13. The QAPP should be clarified that independent pesticide and PCB Aroclor samples are produced during ISM processing, and extracted independently with slightly different procedures.

Response and Evaluation: the QAPP has been modified accordingly; acceptable.

14. Melissa Fuller-Gustavel is reviewing laboratory SOPs listed in the QAPP to ensure that they are the most recent SOPs.

Response and Evaluation: revised SOPs were submitted and the QAPP has been modified accordingly; acceptable.

List of recommendations:

15. The Quality Assurance Manager agreed to discuss how partially frozen samples should be treated at sample check-in at a meeting of all TestAmerica QAMs in early June.

Response and Evaluation: no response received; no action required, although **the validator may choose to ask for further information regarding sample acceptance policies**.

16. The laboratory should have an internal discussion about appropriate safety equipment and procedures in the ISM laboratory for handling samples with potentially low levels of asbestos. A statement or protocol should be submitted to the project team prior to the onset of field sampling.

Response and Evaluation: no response received; no action required. The auditor encourages the laboratory to use appropriate safety equipment and procedures due to the potential presence of asbestos in some project samples, as indicated in the QAPP.

17. Instruments which can be electronically connected to the laboratory information system should be scheduled for upgrades to achieve direct connections, thus reducing both the risk of potential transcription errors and competition for data terminal access.

Response and Evaluation: no response received; no action required.

18. To ensure that project action limits are achieved whenever possible, the laboratory should

be instructed to report "multiple dilutions for the lowest reporting limits" whenever possible.

Response and Evaluation: no response received; **acceptable if this is discussed in a kick-off meeting and documented in a coordination memo**.

19. We have requested a laboratory duplicate to allow independent evaluation of field and laboratory precision; Diane will select which sample will be used for this, and will include one for every parameter.

Response and Evaluation: The QAPP has been updated to include one laboratory duplicate for every 20 ISM samples, which was Diane's suggestion for frequency. **Response acceptable if this is discussed in a kick-off meeting and documented in a coordination memo.**

20. A percent moisture determination on the ISM solid samples will not be needed, except for the routine volatiles percent moisture, which will be sub-sampled prior to drying.

Response and Evaluation: no response received; **acceptable if this is discussed in a kick-off meeting and documented in a coordination memo.**

21. The sub-sample to be used for determination of pH in ISM solids, and the sub-sample to be used for percent moisture for ISM volatiles samples (i.e., in waste piles and sediments) will be taken using ISM procedures from the moist slab cake prior to drying.

Response and Evaluation: noted for QAPP revision; AVS/SEM sub-sample will be taken from the moist slab cake also and this has been incorporated into the QAPP. **Acceptable if all three moist sub-samples are discussed in a kick-off meeting and documented in a coordination memo.**

22. The auditor reiterated that Walkley-Black is the selected method for sediment samples.

Response and Evaluation: QAPP clarified; acceptable.

23. The QAPP should be modified to clarify that hardness should be determined by calculation from the metals results, and the appropriate laboratory SOP should be added to Worksheet #23.

Response and Evaluation: QAPP clarified to show that hardness will only be measured in aqueous samples; acceptable.

24. The auditor instructed Diane to perform equipment blanks at the rate described in the QAPP: one equipment rinse blank should be performed for shared equipment for every 20 decision units, or discrete samples, or per media. Diane noted that this will be incorporated into the internal project protocols.

Response and Evaluation: no response received; **acceptable if this is discussed in a kick-off meeting and documented in a coordination memo.**

25. The hand-written sample weights from the ISM laboratory should be scanned as a permanent record for sub-samples for organics extractions.

Response and Evaluation: discussed during audit closeout and no further response received; **validator should examine laboratory records for proper documentation of sub-sample weights, and the audit will be cited if weights are not properly recorded and reported.**

26. The Johnson Company should ask the project manager to include the option of additional cleanup procedures (silica gel and florisil or alumina column) for interferences (other than sulfur) for the PCB Aroclors analysis.

Response and Evaluation: no response received; **acceptable if this is discussed in a kick-off meeting and documented in a coordination memo.**

27. The QAPP should be updated with the most current laboratory SOPs, based on the laboratory's review of SOP status (see finding #14 above; this is a repeat).

Response and Evaluation: discussed by telephone; acceptable if all SOPs are current and reflect actual laboratory practices.

28. The potential use of reduced volume extraction paired with large volume injection for semivolatiles in porewater should be considered if sample volume is limited, and the QAPP should be modified to allow this as a potential analytical tool.

Response and Evaluation: QAPP revised; acceptable.

29. The validator would appreciate the addition of project-specific control limits to laboratory results reports, to make the data review process more efficient.

Response and Evaluation: discussed by telephone; **acceptable if implemented**.

Appendices

A. References

Quality Assurance Project Plan, Jaite Paper Mill Site Engineering Evaluation/Cost Analysis, Cuyahoga Valley National Park Ohio, The Johnson Company, Inc., May 20, 2016.
TestAmerica-Canton Standard Operating Procedures (SOPs) as presented in the QAPP.
Incremental Sampling Methodology Technical and Regulatory Guidance, Interstate Technology and Regulatory Council, February, 2012.

B. Contact Information

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C. Personnel Interviewed

Amy McCormick, Client Services Representative
Mark Loeb, Quality Assurance Manager
Dorothy Leeson, Quality Assurance
Melissa Fuller-Gustavel, Quality Assurance
Ann Maddux, Sample Receiving Department Manager
Diane Jones, Incremental Sampling Methodology Department Manager
Karen Counts, Inorganics Chemistry Department Manager
Roger Toth, Inorganics Chemistry Department Lead Analyst
Kyle Dillon, Inorganics Chemistry Department Analyst
Alex Colosi, Inorganics Chemistry Department Analyst
Jill Burns, General Chemistry Department Analyst
Julie Sanford, General Chemistry Department Analyst
Tom Harshman, General Chemistry Department Analyst
Gabrielle Renner, General Chemistry Department Analyst
Diem Nguyen, General Chemistry Department Analyst
Olguita Colon, Gas Chromatography Department Manager
Heather Wadsworth, Lead PCBs Analyst
Justin Ross, Organics Extraction Lead Analyst (solids)
Caitlin Scott, Organics Extraction Lead Analyst (waters)
Tom Stiller, Volatiles Department Manager

Tom Hula, Semivolatiles Department Manager

D. Supplemental Documents

a. Proficiency Testing Reports WP0415, HW0715, WP1015, HW0115, WP0416, and HW0116